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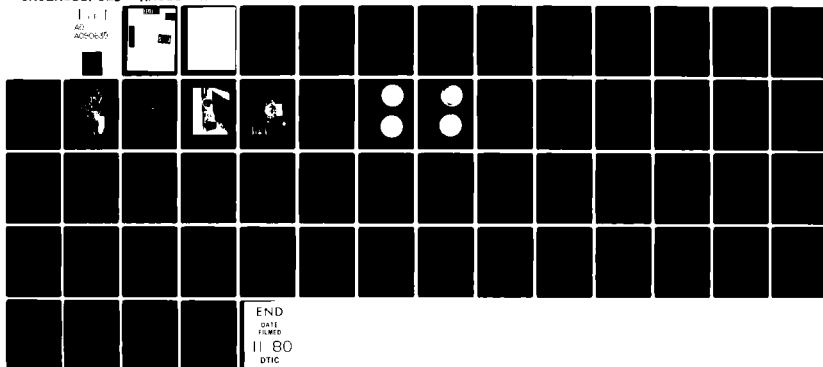
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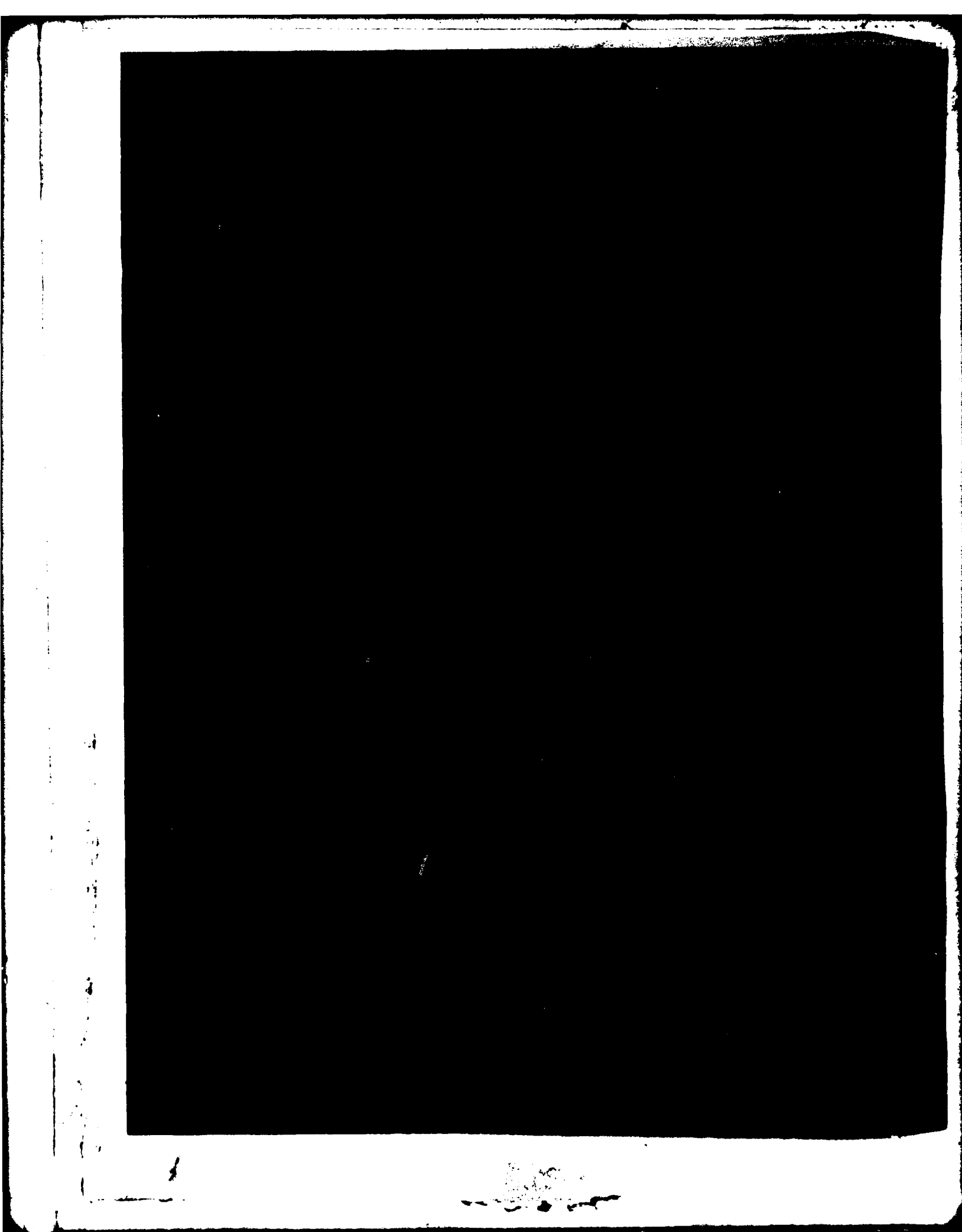
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) An investigative test was conducted at National Space Technology Laboratories (NSTL), Mississippi, from 15 April to 27 April 1979 to provide data on the physical and chemical characteristics of the M446 expelling charge employed in the M259 WP warhead of the 2.75-inch rocket. Numerous warhead malfunctions were recorded during local and proving ground tests. These malfunctions were attributed to incomplete canister ejection from the warhead which resulted in poor smokescreen development. The tests at NSTL compared two lots of expelling charges made to the  (Continued on reverse side)		

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20. ABSTRACT (Contd)

same drawing 5 years apart. New expelling charges functioned satisfactorily; whereas, the old charges performed marginally to poorly. Closed-bomb tests provided convincing data that performance degradation could be expected with shelf life. The new charges developed pressure three times as fast as the old charges although peak levels were not drastically different. Because of the poor consistency of pressure development, high standard deviations resulted. Thus significant differences between the means of old charges and new charges existed at the 0.10 level of significance using the Student's t-distribution for comparison of means. Tests for moisture content and affinity for water were conducted. These tests showed that the charge composition, probably due to the percentage of black powder, was susceptible to water pickup while also containing an appreciable amount of moisture as received in containers specified hermetically sealed. The moisture content aided poor functioning performance. It is recommended that a new composition be developed for the expelling charge which does not degrade with time nor is susceptible to moisture.

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## PREFACE

The investigation described in this report was authorized under Project W31U318324EG04. Work was performed at the Hazards Range Operation Section, Computer Sciences Corporation, National Space Technology Laboratories Station, Mississippi, from 15 April to 27 April 1979.

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## Acknowledgment

The author wishes to thank the personnel of the Hazards Range Operation Section, Computer Sciences Corporation, National Space Technology Laboratories Station, Mississippi, for the cooperation and testing expertise in accomplishing the various phases of this investigation. Particular thanks go to Mr. Fred McIntyre whose efforts as detailed test plan writer, test director, and coordinator of extra-section testing facilitated the smooth progress of the project. Mr. R. Ross is mentioned for his efforts in chemical analyses, the results of which showed very distinct differences between the two lots of propellant under test.

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## SUMMARY

The malfunctioning M259 rocket warheads, as observed during local and proving ground tests, were traced to low functioning reliability of a lot of M446 fuze expelling charges manufactured in November 1973. These charges were replaced by a lot of charges manufactured in October 1978 which performed satisfactorily. The failure of the charge to eject the canister from the warhead casing within the delay time of the burster resulted in canisters functioning inside the casing which, in turn, led to a poorly developed smokescreen. An effort was initiated to determine, by test, the physical and chemical characteristics of the expelling charge with an emphasis on pressure-time relationships.

Those physical characteristics observed were compared to the engineering drawing requirements. There were 168 pellets and 67 of the pellets did not meet these requirements. The results of tests of significance on the means of old and new charges are as follows:

<u>Test</u>	<u>Significantly different at .10 level</u>
Density	No
Autoignition temperature	No
Decomposition temperature	Yes
Heat of combustion	Yes
Open-air burn	Yes
Closed-bomb	
Maximum pressure	Yes
Pressure rise rate	Yes
Burn time	Yes
Ignition delay	Yes
Time to peak pressure	Yes
Gas volume (-50°F, +145°F)	Yes
Gas volume (ambient)	No
Moisture content	Yes

The closed-bomb tests provided the most distinct differences between old and new charges. The threefold rate of pressure rise of the new over the old charges is directly related to the successful ejection of canisters from the warhead casings by new charges.

Although conclusions could not be drawn from the few chemical analyses conducted, observation of the homogeneity of the pellet and the functioning properties of the charges indicated a severe degradation of the constituents with time. Flakes of cellulose nitrate were present on the surface and throughout the old pellets. New pellets did not exhibit this phenomenon. Based on the known performance characteristics of black powder, it is assumed that the presence of this material in the pellet enhances its susceptibility to moisture (i.e., humidity) thus increasing the probability of degraded performance.

It is recommended that the composition of the pellet be changed to improve shelf life and moisture resistance.

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## INVESTIGATION OF PERFORMANCE DEGRADATION OF M446 FUZE EXPELLING CHARGE FOR 2.75-INCH ROCKET WITH M259 WP WARHEAD

### I. PURPOSE.

The purpose of this investigation was to define the physical and chemical characteristics of two lots of the expelling charge, drawing 65C11050, in the M446 fuze employed in the 2.75-inch rocket M259 smokescreening warhead.

### II. BACKGROUND.

The 2.75-inch rocket with M259 smokescreening warhead employs the M446 base detonating fuze which is positioned in the aft end of the warhead casing by a glass-filled Nylon fuze support. The fuze consists of a plastic case, a firing pin and body assembly, a safety and arming device, a delay assembly, and a polyethylene-encased expelling charge. Upon functioning, the 4.5-second delay burns through the plastic to ignite the expelling charge. The pressure buildup which results from the burning acts on an aluminum pusher plate on which the WP-filled canister rests. The piston effect ejects the canister from the warhead casing.

Malfunctioning warheads, observed during local and proving ground tests, have indicated failure of the canisters to be ejected from warhead casing. Local tests have also resulted in partial ejections. In both instances, ignition of the burster assembly by the expelling charge causes the canister to rupture while it is still in the warhead. Failure of the fuze to eject the canister can be observed since the round impacts approximately 1500 meters longer than normal functioning rounds.

In order to improve the functioning reliability, a more recent lot of expelling charges was retrofitted into M446 fuzes. The date of manufacture of the "new" charges was October 1978. The "old" charges were manufactured in November 1973. Improved fuze functioning was observed. At this time, it was conjectured that the pressure being generated during burning of the "old" charge was insufficient to push the canister out of the warhead casing.

The tests conducted at the National Space Technology Laboratory, NSTL, by the Hazards Range Operations Section, Computer Sciences Corporation, NSTL Station, Mississippi, were primarily directed at pressure-time measurements. A charge which developed sufficient pressure over too long a period was of no value if the time delay in the burster assembly was exceeded. This situation caused the canister to burst prior to complete ejection from the warhead casing, which resulted in a poorly developed smokescreen. Additional tests were conducted to more thoroughly define the expelling charge characteristics.

### III. DATA ACQUISITION PROCEDURE.

A total of 86 old and 84 new charges was submitted to the test agency. All charges were encased in a hermetically sealed polyethylene container. Old charges had a 0.5-inch hole in the output side of this container.

Temperature conditioning was accomplished for a minimum of 8 hours to assure equilibrium had been attained in each pellet prior to testing. Each sample was maintained at the conditioning temperature until it was fired to provide a more accurate evaluation of the effects of temperature on performance.

The engineering drawing and specifications of the expelling charge were provided to the test agency.

The tests described below were performed on both old and new charges.

All tests for significance were applied using a two-tailed distribution with a 90% confidence interval. Assuming all charges were made under the same process conditions, the Student's t-distribution for comparing means with equal standard deviations was applied using a 0.10 probability.

A. Initial Inspection.

A visual inspection of each sample was conducted upon receipt of material. Each charge was weighed as received (i.e., in polyethylene containers) and assigned a sample number. All charges were also weighed without the polyethylene container. In the old charges, the pellet was taken from the plastic container through the hole. The seal of the plastic container of new charges was broken; pellets were weighed and replaced in the plastic cup. The cup was reheat-sealed. This operation was accomplished in the same day to avoid absorption of humidity.

B. Density Determination.

Each charge was weighed to the nearest 0.001 gram and measured for volume calculations. The density was calculated as the mass divided by the volume.

C. Autoignition Temperature.

The autoignition temperature test determined the temperature at which the material reacted when it liberated heat due to self-heating. The sample, ground into fine powder and with a thermocouple imbedded in it, was placed in an automatically controlled oven. The oven was heated at a controlled rate until the sample began to liberate heat, as indicated by a strip chart recorder. At this point, the oven temperature was maintained constant until the sample reacted rapidly at its autoignition temperature. Multiple specimens taken from various areas of the same charge were tested.

D. Decomposition Temperature.

The decomposition temperature test determined the ignition temperature and other physical and chemical reactions which may occur in a pyrotechnic mixture upon heating. The sample configuration was a fine powder. Iterations of this test using various areas of the same expelling charge were conducted. The test was conducted in a 70°F environment. The measurement recorded was the temperature difference between the sample pyrotechnic and a thermally

inert reference material as both were heated simultaneously at a constant rate of temperature increase. Any endothermic or exothermic changes in the sample were recorded on a strip chart. These changes may be related to dehydration, decomposition, crystalline transition, melting, boiling, vaporization, polymerization, oxidation, or reduction. The point at which the maximum temperature differential occurs between the sample and reference was the reported decomposition temperature.

E. Heat of Combustion.

The heat of combustion test determined gross heat content in terms of calories per gram of expelling charge. The test method employed a Parr bomb calorimeter. A 1-gram sample was placed in an oxygen-filled (5 atmospheres) standard calorimeter bomb which was submerged in water. The rise in water temperature which resulted from burning the sample was recorded. Standardization of the Parr bomb was done with standard benzoic acid in accordance with ASTM D240-64. Expelling-charge samples were also conducted in accordance with this method. Three new charges and three old charges were tested.

F. Hygroscopicity.

The hygroscopicity test determined the amount of moisture the sample absorbed at 75% humidity. The sample was prepared by completely sieving a pellet through a 50-mesh screen onto a 100-mesh screen. Samples remained in the atmosphere until moisture equilibrium was attained. After conditioning sample weights were determined, the percentage of moisture absorbed was calculated as

$$\frac{B - A}{A} \times 100$$

where:

A = weight of sample before conditioning  
B = weight of sample after conditioning

G. Thermal Stability.

The thermal stability test was conducted to determine whether the material experienced a change in state during conditioning in an explosion-proof oven at 75°C for 48 hours. The charges were weighed without the polyethylene cup, returned to the cup, and placed into conditioning. They were weighed after conditioning without the cup. The change in weight was calculated. Visual observations of change in configuration were made. The oven temperature was continuously monitored throughout the conditioning period.

H. Weight Loss.

The weight loss test was conducted to determine the moisture and volatile content of the sample material. The polyethylene bags were removed from the samples which were weighed before and after each conditioning sequence. The sequences were: (1) 4 hours' soak at 50°C and

18 inches of vacuum, (2) 66 hours' exposure at ambient temperature and humidity, and (3) 48 hours' soak at 50°C and 18 inches of vacuum. Two samples of each lot of old and new charges were subjected to this test. The percent weight loss was calculated as

$$\frac{B - A}{A} \times 100$$

where:

A = weight before conditioning, grams

B = weight after conditioning, grams

I. Electrical Spark Test.

The electrical spark test was conducted to determine the sensitivity of the expelling charge. The measurement was made in terms of the minimum amount of energy, expressed in joules, in an electrical spark discharge required to ignite the sample. The test quantity was placed on a grounded anode and the electrode, which was charged with high voltage through a series capacitor, was lowered to the anode until an electrical spark appeared. The energy level was increased to 50 joules; reactions were observed. The energy was calculated as  $\frac{1}{2} CV^2$ , where C = capacitance in microfarads and V = voltage in volts. One sample from each of the old and new charges was tested in this manner. Ten trials from each sample were conducted in an environment of 75°F and 55% relative humidity.

J. Friction Pendulum Test.

The friction pendulum test was conducted to determine whether the test material was susceptible to frictional forces. Ten trials with samples from the old charges and ten from the new charge were conducted using the steel shoe. No trials were conducted with the fiber shoe. All tests were conducted on ambient-conditioned samples.

K. Impact Sensitivity.

The impact sensitivity test was conducted to determine the sensitivity of the test material to mechanical shock (impact). The Bureau of Mines impact apparatus was used to determine the "go, no-go" capacity of the test material. A series of twenty tests was performed on 10-mg samples from one old expelling charge and one new charge. Each sample, consisting of two or three chunks of material broken off a pellet, was placed in a test cup and a 2-kg test weight was dropped onto the material from heights of 9.53 cm (3.75 in) and 25.4 cm (10 in) in separate tests. Following these tests, additional trials were conducted to determine the minimum drop height at which a reaction occurred.

L. Open-Air Burn.

The open-air burn test was conducted to observe the burning characteristics of the material under test, while it was in an unconfined state. Each trial was conducted on a complete pellet encased in the polyethylene cup. A small hole was cut in the plastic of each new charge prior to

firing to accept the electrical match. No additional holes were added to the containers of old expelling charges. Tests were conducted on charges conditioned hot [average, 60°C (140°F)], ambient [average, 24°C (75°F)], and cold [-46°C (-50°F)]. Burn time was determined using a stopwatch and was defined as the elapsed time from pellet ignition to consumption. Smoldering particles were not included in the measurement.

#### M. Closed-Bomb Pressure-Time Tests.

The closed-bomb pressure-time tests were conducted in a 22-quart instrumented closed chamber, figures 1 and 2, to determine static pressure, rate of pressure rise, total burn time, maximum temperature attained, gas volume, and ignition delay. The chamber was instrumented with pairs of BLH strain-gage-type transducers, thermocouples, a photoelectric cell, and a firing circuit.

Each test charge was placed on a small pedestal inside the chamber, figure 3. A wooden disk, into which the electric match was positioned, was placed over the test charge and the assembly was taped lightly to the pedestal, figure 4. All firing was done remotely through a panel which controlled the sequential operations of the instrumentation.

The old charges were fired in the configuration as received. A small hole was cut in the plastic cup of the new charges, however, to accept the spit from the electric match. Samples of new and old charges were temperature conditioned at -46°C (-50°F), 23.8°C (75°F), and 62°C (144°F) for a minimum of 8 hours prior to firing. An attempt was made to minimize the time between removal of each sample from conditioning to firing; however, as much as 5 minutes occasionally elapsed before firing.

Representative still photographs were taken before and after selected samples.

The information in table 1 applies to figure 2, the closed bomb:

Table 1. Instrumentation of Closed-Bomb Test

Channel	Type measurement	Range	Recorder
1	Static pressure	0-20 psi	Oscillograph
2	Static pressure	0-20 psi	Oscillograph
3	Photocell	0-5 Volts	Oscillograph
4	Internal temperature	Ambient, 600°F	Oscillograph
5	External temperature	Ambient, 600°F	Oscillograph
6	External temperature	Ambient, 600°F	Oscillograph
7	Timing temperature	1-Millisecond intervals	Oscillograph

Using the weight of each test charge and the recorded maximum temperature obtained inside the chamber, the volume of gas evolved was calculated. All other measurements were reduced from the strip charts.

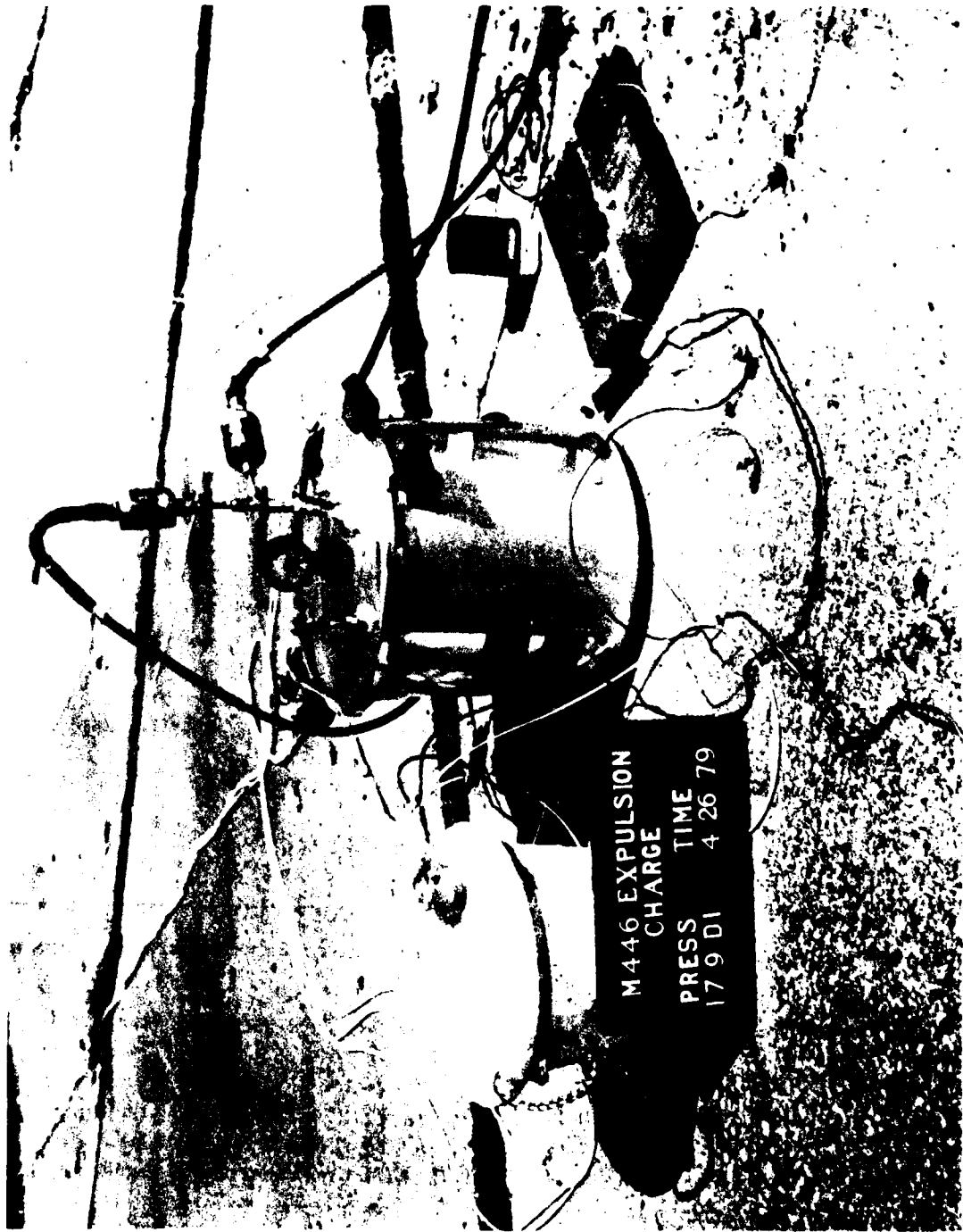


Figure 1. Instrumented Closed-Bomb Setup

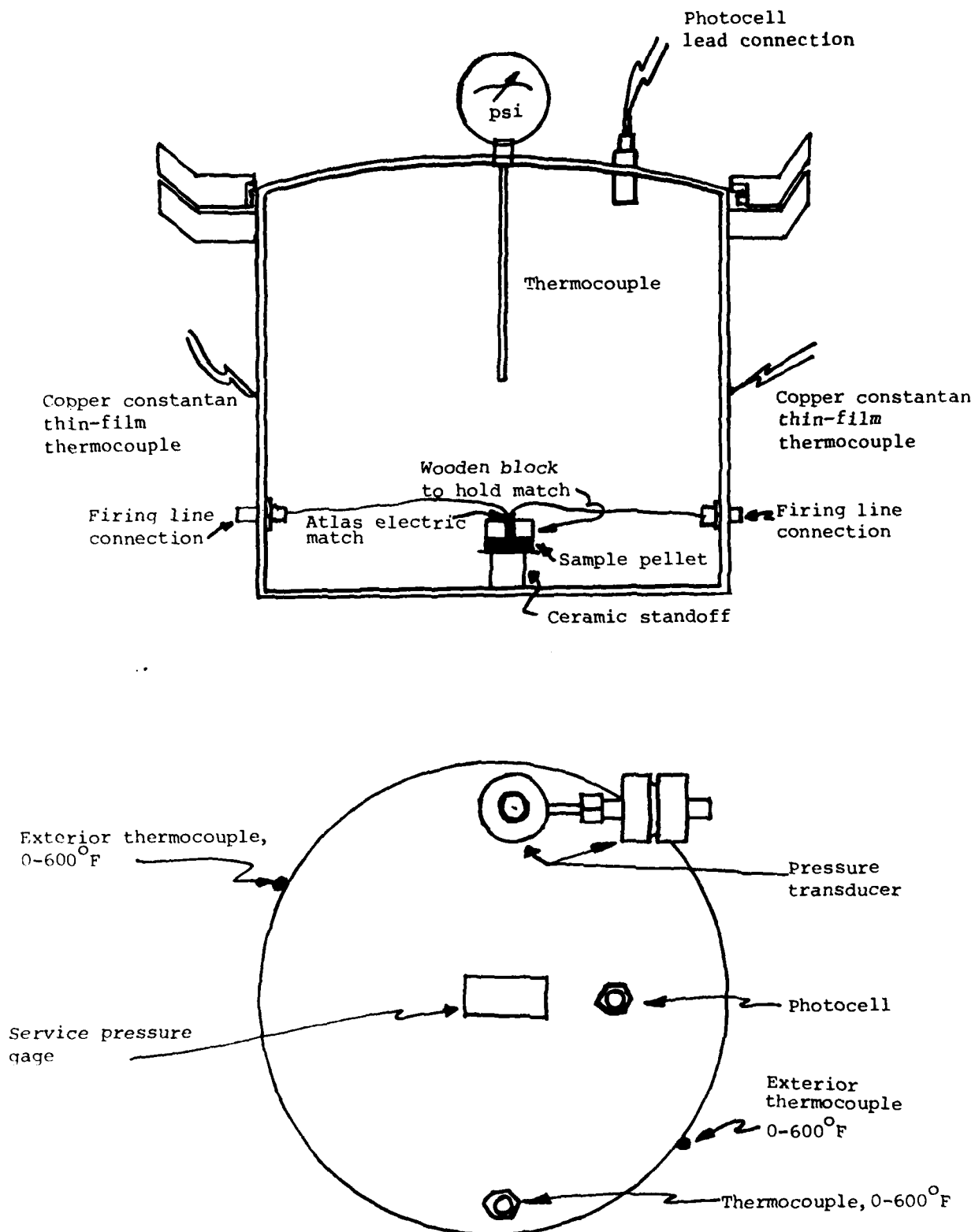


Figure 2. Schematic of Instrumented Closed Bomb





Figure 3. Closed-Bomb Chamber with Pedestal Where Test Pellets Were Fired



Figure 4. Closed-Bomb Chamber with Test Pellet in Place on Pedestal

#### N. Moisture Content.

The Karl Fischer titration was the analytical method employed for determining moisture content. Five of the six old and new charges tested for heat of combustion were analyzed for moisture. The 1.5-gram samples were analyzed in bulk granular form. The test was conducted under environmental conditions of 23°C (74°F) air temperature and 44% relative humidity. The samples were soaked in the extraction solvent of 70% chloroform and 30% methanol for 3 minutes prior to titration. The moisture measurements are based on the Karl Fischer titration in which the Karl Fischer (KF) reagent reacts quantitatively with the water in the sample which produces a chemical change (titration end point) detected amperometrically. A Beckman KF4B aquameter was used in these trials. Moisture content was calculated as H<sub>2</sub>O in parts per million molar

$$\frac{CF \times 10^6}{W}$$

where

C = KF reagent required to reach end point, milliliters

F = water equivalence of KF reagent, milligrams per milliliter

W = weight of sample, milligrams

10<sup>6</sup> = conversion factor for parts per million

#### IV. RESULTS.\*

##### A. Initial Inspection.

New charges were uniform in appearance. The polyethylene container was sealed completely in all instances. All new charges were similar. The old charges were not uniform throughout any given pellet or from one pellet to another. The existence of shiny flecks of material was prevalent in old charges, figures 5 and 6. When physically extracted from the pellet, this material was determined by solubility tests to be cellulose nitrate.

The individual pellet weights, less the polyethylene cup, were compared against the value appearing on drawing 65C11050,\*\* that value being 5.9 ± 0.4 grams, prior to sealing in the cup. A total of 31 of 84 old pellets and 36 of 84 new pellets weighed less than the weight shown on the drawing.

##### B. Density Determination.

The results of this phase are in table 2.

Table 2. Summary of Results of Density Determination

Charge type	Number of samples	Weight of pellet		Pellet density	
		Mean	Standard deviation	Mean	Standard deviation
		gm		gm/cc	
Old	84	5.5549	0.2027	1.63	0.06
New	84	5.5375	0.1661	1.63	0.06

Old and new pellets are statistically comparable with respect to weight and density.

\* Note: Round-by-round data are presented as appendix A.

\*\* Drawing appears in appendix B.

NEW

OLD

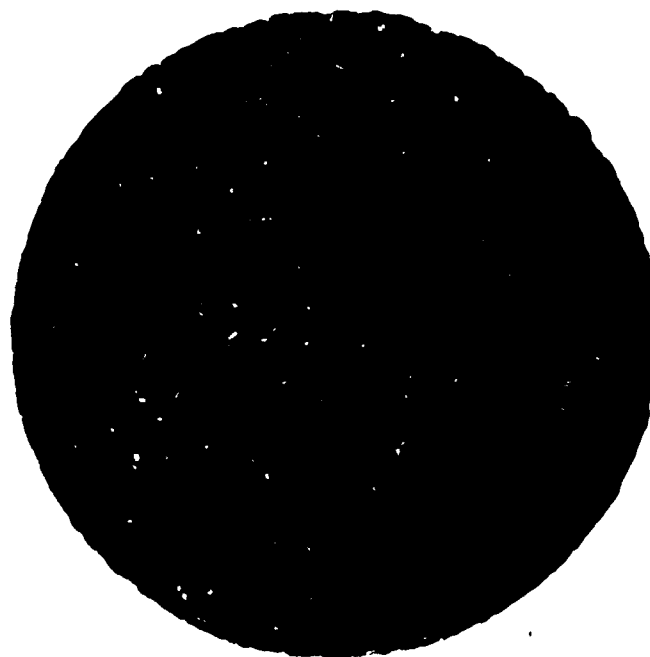
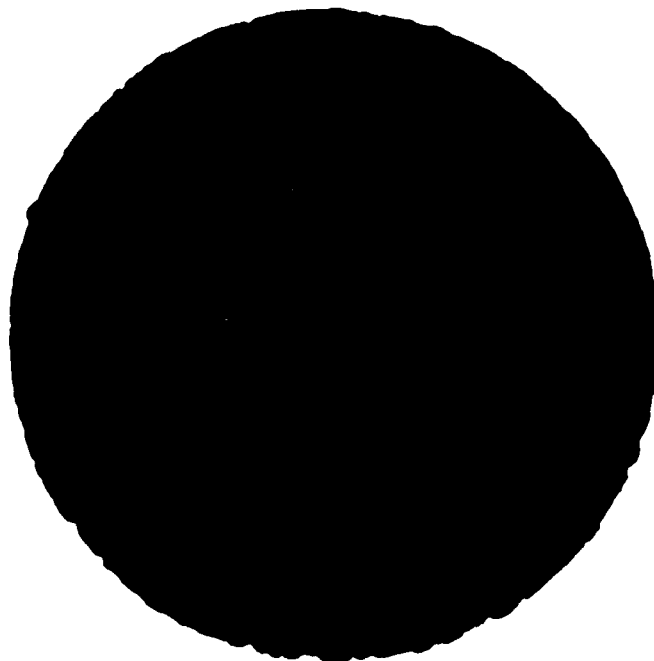
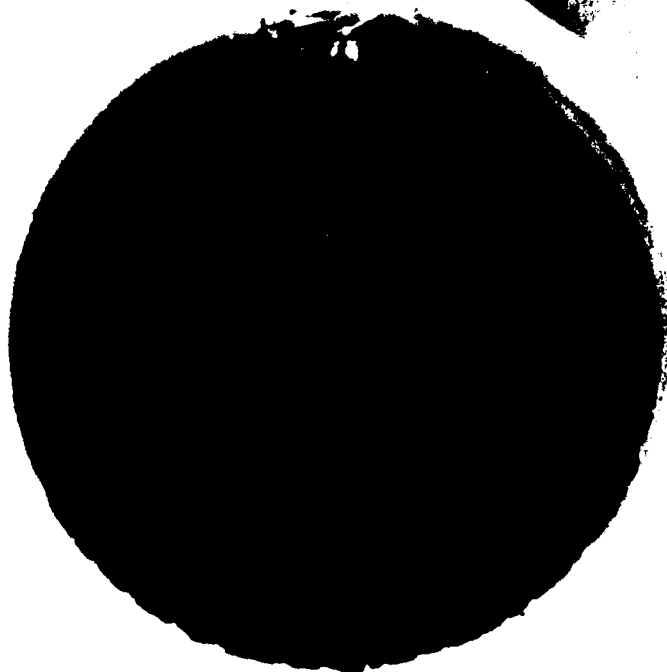


Figure 5. Visual Comparison of Old and New Charges. Note the presence of white flecks of cellulose nitrate randomly scattered throughout the old charge.

NEW



OLD



Figure 6. Visual Comparison of Expelling Charges as Received. Note the presence of white flecks of cellulose nitrate randomly scattered throughout the old charge.

C. Autoignition Temperature.

The average of five trials for the autoignition temperature of the new material was 2°C less than the average of four trials of the old material. The standard deviation of the new material was also 1.8°C less than that of the old material. Five samples were tested from each expelling charge; however, one trial of the old material was not included due to a poor test. There was no significant difference at the .10 level between the means of the autoignition temperature of the old and new expelling charges.

D. Decomposition Temperature.

A summary of the results of decomposition and autoignition temperature tests is found in table 3.

Table 3. Summary of Results of Decomposition and Autoignition Temperature Tests

Charge type	Number of samples	Decomposition temperature		Number of samples	Autoignition temperature	
		Mean	Standard deviation		Mean	Standard deviation
		°C			°C	
Old	5	164	12.7	4	147	7.8
New	5	156	5.0	5	145	6.0

There was a significant difference at the .10 level between the means of the decomposition temperature of the old and new expelling charges.

E. Heat of Combustion.

Samples of an approximate weight of 1 gram were taken from three old charges and three new charges. The average heat output of the new charges, 2941 cal/gm, was approximately 50% greater than the average heat output of the old charges which was 1984 cal/gm. Table 4 summarizes these results. The standard deviation of the new charges was 4.2 cal/gm, whereas that of the old charges was 72.9 cal/gm.

Table 4. Results of Parr Bomb Calorimeter Tests

Sample No.	Charge type	Charge weight	Increase in water temperature	Heat of combustion
		gm	°C	cal/gm
20	Old	1.0070	0.762	1885.4
23	Old	1.0192	0.821	2007.02
99A	Old	1.0115	0.828	2059.54
72	New	1.0163	1.202	2946.8
73	New	1.0153	1.198	2939.9
74	New	1.0121	1.193	2936.8

There is a significant difference at the .10 level between the means of the heat of combustion of the old and new expelling charges.

F. Hygroscopicity.

The old charges had a slightly greater affinity for moisture than did the new charges. The old samples absorbed 0.52% additional weight in water at 75% humidity, whereas the new samples absorbed 0.36% weight in water at the same atmospheric conditions.

G. Thermal Stability.

There was no visual change in state or configuration of either sample after a 48-hour exposure at 75°C. The old sample had an initial density of 1.64 gm/cc. The change in weight due to the exposure was 6.51%. The new sample had an initial density of 1.63 gm/cc. The change in weight due to heat exposure was 5.34%.

H. Weight Loss.

After the initial exposure to heat and vacuum, the old charges lost more moisture/volatiles than the new charges. These pellets also gained more moisture during exposure to ambient temperature and humidity. However, after the long-term 48-hour soak at 50°C and 18 inches of vacuum, the new pellets lost more weight. The data are in table 5. Both old and new pellets experienced a considerable change in weight.

Table 5. Summary of Results of Weight-Loss Test

Charge type	Sample No.	Initial weight	Weight after 4-hr soak at 50°C and 18 inches of vacuum	Weight after 66-hr soak at ambient temperature and RH	Weight after 48-hr soak at 50°C and 18 inches of vacuum	Total weight loss
		gm	gm (% charge)	gm (% charge)	gm (% charge)	%
Old	5	5.2732	5.2319 (-0.78)	5.2575 (+0.49)	5.1819 (-1.44)	1.73
	26	5.7860	5.7385 (-0.82)	5.7700 (+0.55)	5.6773 (-1.61)	1.88
New	2	5.2880	5.2537 (-0.65)	5.2742 (+0.39)	5.1817 (-1.75)	2.01
	31	5.7363	5.7016 (-0.60)	5.7204 (+0.33)	5.6276 (-1.62)	1.89

I. Electrical Spark Test.

Neither the old nor the new expelling charges were sensitive to electrical spark at energy levels from 0 to 50 joules. In all trials, the capacitance was maintained constant at 1 microfarad ( $\mu\text{f}$ ), whereas the voltage was increased from 100 to 10,000 volts. The energy was calculated as  $\frac{1}{2}CV^2$ . There was no reaction from any sample at any energy level.

J. Friction Pendulum Test.

There were no reactions in 10 trials with the steel shoe on each of the old and new materials. Tests with the fiber shoe were not conducted because of the nonreactivity of the samples to the steel shoe.

#### K. Impact Sensitivity.

Ten trials were conducted on each of the old and new pellets at a drop height of 10 inches. In all cases, the sample exploded. Ten trials of each pellet were again conducted at a drop height of 3.75 inches. The old pellet samples exploded in four cases, decomposed in two cases, and did not react in four cases. The new pellets exploded in eight cases and did not react in two cases. The area of the pellet from which each sample was taken was not recorded. The minimum drop height at which no reaction occurs is 6.35 cm (2.5 in) for the old material, which corresponds to an energy of 1.49 joules, and 5.72 cm (2.25 in) for the new material, which corresponds to an energy of 1.12 joules.

#### L. Open-Air Burn.

In all cases, the new expelling charges burned twice as fast as the old charges. After hot conditioning, the mean burn times of both new and old charges were less than the respective mean burn times after ambient conditioning. Table 6 summarizes the results.

Table 6. Summary of Results of Open-Air Burn

Charge type	Number of samples	Conditioning temperature	Burn time		Pellet weight		Pellet density	
			Mean	Standard deviation	Mean	Standard deviation	Mean	Standard deviation
		°F		sec		gm		gm/cc
Old	8	Ambient (75)	1.31	0.28	5.5255	0.203	1.62	0.06
New	8	Ambient (75)	0.78	0.25	5.5103	0.196	1.62	0.06
Old	5	+145	1.0	0.13	5.3448	0.306	1.57	0.09
New	5	+145	0.52	0.20	5.5490	0.143	1.63	0.04
Old	5	-50	1.8	0.30	5.5422	0.233	1.63	0.07
New	5	-50	0.8	0.62	5.5299	0.228	1.63	0.07

There is a significant difference at the .10 level between the mean burn time of the old and new charges at all temperatures.

#### M. Closed-Bomb Pressure-Time Tests.

There was a significant difference between old and new charges at the three test temperatures during the measurements of maximum pressure, rate of pressure rise, total burn time, and ignition delay. There was a significant difference in the volume of gas evolved at -50°F and +145°F.

The rate of pressure rise of the new charges was three times that of the old charges at the three test temperatures. The ignition delay and total burn time of the old charges were each longer than those of the new charges by a factor of two at the three test temperatures. The time to peak pressure of the old material was longer than that of the new material by a factor of 2.5 at the three temperatures. Characteristic pressure-time traces of an old and new charge are shown in figure 7, A, and 7, B, respectively. The results of the closed-bomb tests are summarized in table 7.



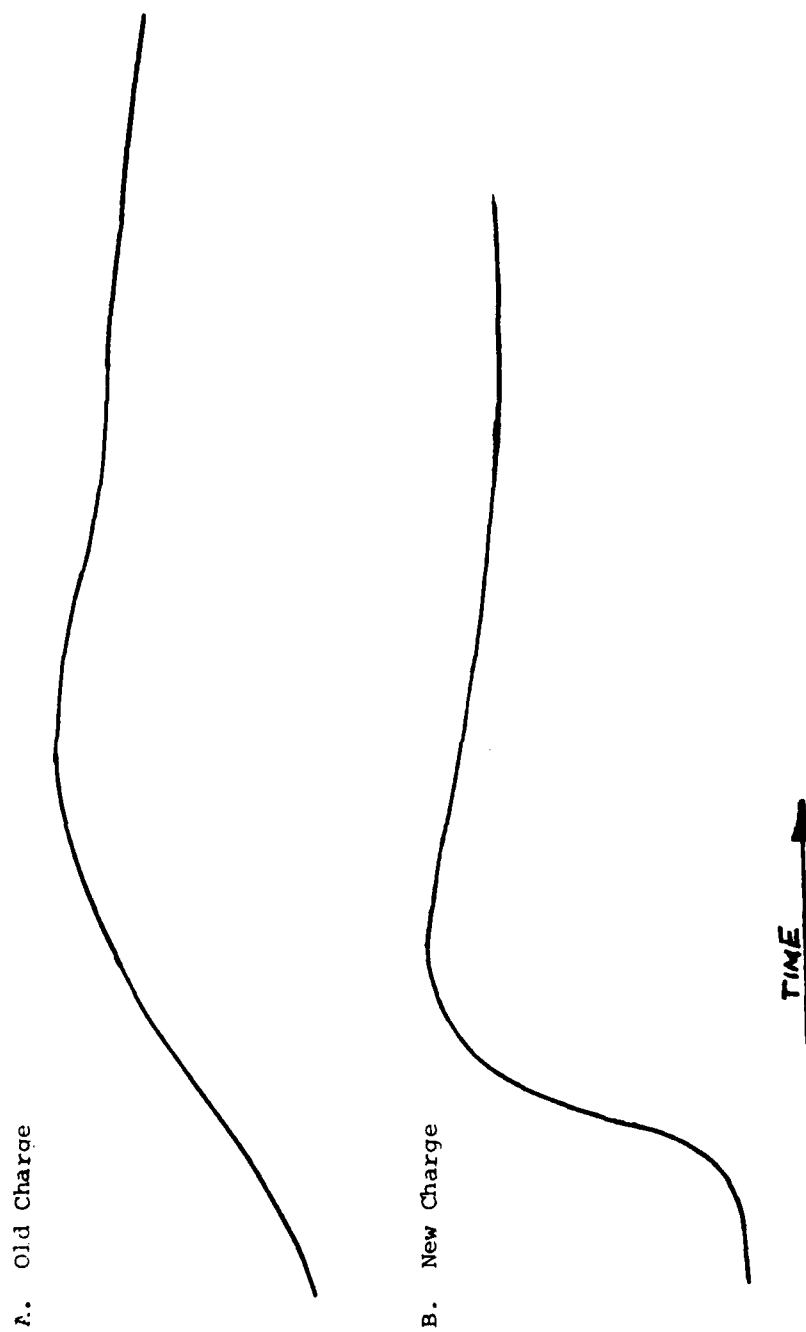


Figure 7. Characteristic Pressure-Time Traces of Old (A) and New (B) Expelling Charges.  
Note rapid rate of rise of new (B) charge in comparison to old (A) charge.

Table 7. Summary of Results of Closed-Bomb Tests

	-50°F (-45.6°C)			Ambient (23.8°C)			+145°F (62°C)		
	Old	New	t*	Old	New	t*	Old	New	t*
Weight, gm									
Mean	5.3711	5.5301	No	5.6304	5.6312	No	5.6740	5.5033	No
Standard deviation	0.22	0.16		0.130	0.142		0.174	0.122	
Density, gm/cc									
Mean	1.57	1.62	No	1.65	1.65	No	1.67	1.61	Yes
Standard deviation	0.06	0.05		0.04	0.04		0.05	0.04	
Maximum pressure, psi									
Mean	46.04	56.80	Yes	50.98	60.72	Yes	57.54	73.75	Yes
Standard deviation	1.14	1.40		1.98	4.03		7.87	4.05	
Pressure rise rate, psi/sec									
Mean	65.18	230	Yes	79.34	251.63	Yes	108.84	349.4	Yes
Standard deviation	16.4	32.7		11.59	82.06		23.79	71.14	
Maximum temperature, °C									
Mean	293	300	No	242 <sup>+</sup>	349	Yes	346	228	Yes
Standard deviation	91	21		73.4 <sup>+</sup>	35.5		53.6	27.8	
Gas volume, l/gm									
Mean	2.57	2.05	Yes	2.02 <sup>+</sup>	2.04	No	2.15	1.38	Yes
Standard deviation	0.43	0.04		0.35 <sup>+</sup>	0.05		0.32	0.09	
Burn time, sec									
Mean	1.02	0.59	Yes	0.95	0.52	Yes	0.61	0.34	Yes
Standard deviation	0.34	0.14		0.31	0.10		0.13	0.08	
Ignition delay, ms									
Mean	218	86	Yes	146	75	Yes	172	47	Yes
Standard deviation	135	15		66.9	23		37	11.3	
Time to pressure peak, ms									
Mean	748	253	Yes	662	269	Yes	537	218	Yes
Standard deviation	213	34.3		99.9	87.25		59.3	37.5	
Number of samples	5	5		5	5		5	5	
<sup>+</sup> Number of samples				4					

\* Significant difference at 0.10 level using two-tailed t-test.

#### N. Moisture Content.

The average moisture content in the three old samples was more than 2.5 times the average moisture content of the three new samples. The results are found in table 8.

Table 8. Results of Moisture Content Determination

Charge type	Sample No.	Weight of sample	KF reagent	H <sub>2</sub> O content
		mg	ml	molar ppm
Old	33	1001.3	2.87	$4.38 \times 10^3$
	23	1019.2	3.62	$5.43 \times 10^3$
	99A	1011.5	3.75	$5.67 \times 10^3$
New	72	1016.3	1.53	$2.30 \times 10^3$
	73	1014.2	1.38	$2.08 \times 10^3$
	74	1012.1	1.02	$1.54 \times 10^3$

There is a significant difference at the 10% level between the means of the moisture content of old and new expelling charges.

#### V. ANALYSIS.

Malfunctions experienced during M259 warhead firings which were attributed to the expelling charge can be substantiated by the closed-bomb data. The rate of pressure rise and the maximum pressure developed directly influence the time required for complete ejection of the canister from the warhead prior to its burst. This time was designed to be no more than 30 milliseconds. Included in this pressure-time relationship is the time required for the expelling charge to develop sufficient pressure to shear the six pins retaining the nose cone.

Assuming the available volume in the round to be 5 in<sup>3</sup> and showing the closed bomb to be 1342 in<sup>3</sup>, the calculations of table 9 were made:

Table 9. Time Required to Shear Six Nose-Cone Pins

Temperature	Time required to shear six pins		Rate of pressure rise in closed bomb	
	Old	New	Old	New
°F	ms		psi/sec	
-50	13.2	3.7	65.2	230
+70	10.8	3.4	79.3	251.6
+145	7.9	2.5	108.8	349.4

The old expelling charges are three times as slow as the new expelling charges in developing sufficient pressure to shear the six nose-cone pins. Velocity measurements by photographic methods of the

canister exiting the warhead have shown this value to be approximately 15 feet per second. Based on this velocity and a burster delay of 150 milliseconds, a canister will function 1.2 feet outside the warhead casing with the new expelling charge. Because the velocity of an ejecting canister is not obtainable when an old expelling charge is used, an assumption was made to complete this analysis. This assumption is that the velocity of the canister with an old charge is one-third the velocity which results when a new charge is used. This assumption is based on the observed rate of pressure rise each type of charge exhibited during closed-bomb tests. Based on this assumption, a canister would require 10.8 milliseconds to shear the nose-cone pins and 210 milliseconds to clear the warhead casing, a total of 220.8 milliseconds. With a delay time of 150 milliseconds, failures of the type where canisters burst prior to complete ejection from the warhead casing can be expected.

#### VI. CONCLUSIONS.

It is concluded that:

1. The new expelling charges functioned better than the old charges.
2. Moisture substantially affected the performance of both old and new charges.

#### VII. RECOMMENDATIONS.

It is recommended that:

1. The current expelling charge be replaced by a pellet, configured (1) to generate sufficient pressure at all temperatures to expel the canister and (2) not to degrade within the shelf life of the complete round.
2. The burster delay time be increased to a mean of 250 milliseconds, since it has been calculated that no adverse effect on smokescreen development results.

APPENDIX A  
ROUND-BY-ROUND DATA

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## INITIAL INSPECTION

### Visual Inspection of New Material

Sample No.		Sample No.	
1.	Dull both sides	43.	Dull both sides
2.	Dull both sides	44.	Dull both sides
3.	Dull both sides	45.	Dull both sides
4.	Dull both sides with some shiny specks	46.	Dull both sides
5.	Dull both sides with some shiny specks	47.	Dull both sides
6.	Dull both sides	48.	Dull both sides
7.	Dull both sides	49.	Dull both sides
8.	Dull both sides	50.	Dull both sides
9.	Dull both sides	51.	Dull both sides
10.	Dull both sides	52.	Dull both sides
11.	Dull both sides	53.	Shiny both sides
12.	Dull both sides	54.	Dull both sides
13.	Dull both sides	55.	Shiny both sides
14.	Dull both sides	56.	Dull both sides
15.	Dull both sides	57.	Shiny both sides
16.	Dull both sides	58.	Shiny both sides
17.	Dull both sides	59.	Dull both sides
18.	Dull both sides	60.	Dull both sides
19.	Dull both sides	61.	Dull both sides
20.	Dull both sides	62.	Dull both sides
21.	Dull both sides	63.	Dull both sides
22.	Dull both sides	64.	Dull both sides
23.	Dull both sides	65.	Shiny both sides
24.	Dull both sides	66.	Shiny both sides
25.	Dull both sides	67.	Shiny both sides
26.	Dull both sides	68.	Dull both sides
27.	Dull both sides	69.	Shiny both sides
28.	Dull both sides	70.	Dull both sides
29.	Dull both sides	71.	Dull both sides
30.	Dull both sides	72.	Dull both sides
31.	Dull both sides	73.	Dull both sides
32.	Dull both sides	74.	Dull both sides
33.	Dull both sides	75.	Shiny both sides
34.	Dull both sides	76.	Shiny both sides
35.	Dull both sides	77.	Dull both sides
36.	Dull both sides	78.	Shiny both sides
37.	Dull both sides	79.	Shiny both sides
38.	Dull both sides	80.	Dull both sides
39.	Dull both sides	81.	Dull both sides
40.	Dull both sides	82.	Dull both sides
41.	Dull both sides	83.	Dull both sides
42.	Dull both sides	84.	Dull both sides

## Visual Inspection of Old Material

Sample  
No.

1. Dull on both sides
2. Dull on both sides
3. Dull on both sides but dullest on side with opening
4. Shiny on side opposite opening; dull on open side; also looks like some water spots
5. Dull on both sides; some discoloration on edges of pellets
6. Dull on both sides
7. Dull on both sides
8. Dull on both sides but dullest on opening side
9. Some discoloration on open side; shiny on opposite side
10. Dull with some water spots and discoloration on opening side
11. Dull generally, but some shine on both sides
12. Dull with some shine on both sides
13. Dull on both sides
14. Dull on both sides
15. Dull on both sides
16. Shiny and black on opposite side; dull on opening side
17. Dull on both sides
18. Dull on both sides
19. Dull on both sides
20. Dull on both sides
21. Some shiny specks on both sides
22. Dull on both sides
23. Dull on both sides
24. Dull on both sides
25. Shiny specks on opposite side; dull on opening side
26. Shiny and black on both sides
27. Some specks on both sides
28. Some shiny specks on both sides
29. Shiny both sides
30. Shiny specks both sides
31. Shiny specks both sides
32. Primarily dull with some shiny specks both sides
33. Dull both sides
34. Dull both sides
35. Several shiny specks on both sides
36. Dull both sides
37. Dull both sides
38. Dull front; shiny back
39. Front dull; back shiny
40. Front dull; back shiny
41. Front dull; back shiny
42. Front dull; back shiny
43. Front dull; back shiny
44. Front dull; back shiny
45. Front dull; back shiny
46. Front dull; back shiny
47. Front dull; back shiny
48. Front dull; back shiny
49. Front dull; back shiny

### Visual Inspection of Old Material (Contd)

Sample  
No.

- 50. Dull both sides; back shiny
- 51. Front dull; back shiny
- 52. Front dull; back shiny
- 53. Front dull; back shiny
- 54. Front dull; back shiny
- 55. Front dull; back shiny
- 56. Front dull; back shiny
- 57. Front dull; back shiny
- 58. Front dull; back shiny
- 59. Front dull; back shiny
- 60. Front dull; back shiny
- 61. Front dull; back shiny
- 62. Front dull; back shiny
- 63. Front dull; back shiny
- 64. Front dull; back shiny
- 65. Shiny both sides
- 66. Front dull; back shiny
- 67. Front dull; back shiny
- 68. Front dull; back shiny
- 69. Front dull; back shiny
- 70. Front dull; back shiny
- 71. Front dull; back shiny
- 72. Front dull; back shiny
- 73. Front dull; back shiny
- 74. Front dull; back shiny
- 75. Front dull; back shiny
- 76. Front dull; back shiny
- 77. Shiny both sides
- 78. Front dull; back shiny
- 79. Front dull; back shiny
- 80. Dull both sides
- 81. Dull both sides
- 82. Dull both sides
- 83. Dull both sides
- 84. Dull both sides
- 85. Dull both sides



# DENSITY DETERMINATION

Old material						New material					
Sample No.	Pellet weight	Density	Sample No.	Pellet weight	Density	Sample No.	Pellet weight	Density	Sample No.	Pellet weight	Density
	gm	gm/cm <sup>3</sup>		gm	gm/cm <sup>3</sup>		gm	gm/cm <sup>3</sup>		gm	gm/cm <sup>3</sup>
1	5.6425	1.66	43	5.2820	1.55	1	5.3710	1.58	43	5.2494	1.54
2	5.5261	1.62	44	5.5214	1.62	2	5.2880	1.55	44	5.5341	1.63
3	5.6592	1.66	45	5.7782	1.70	3	5.4482	1.60	45	5.7935	1.70
4	5.8154	1.71	46	5.3472	1.57	4	5.6362	1.66	46	5.3453	1.57
5	5.2732	1.55	47	5.2159	1.53	5	5.7527	1.69	47	5.3997	1.59
6	5.7038	1.68	48	5.5017	1.62	6	5.5634	1.63	48	5.3555	1.57
7	5.6872	1.67	49	5.5009	1.62	7	5.7645	1.69	49	5.5773	1.64
8	5.6338	1.66	50	5.5080	1.62	8	5.6316	1.65	50	5.6112	1.65
9	5.4280	1.59	51	5.7702	1.70	9	5.5915	1.64	51	5.4438	1.60
10	5.5742	1.64	52	5.7356	1.69	10	5.3857	1.58	52	5.2669	1.55
11	4.8269	1.42	53	5.6858	1.67	11	5.4438	1.60	53	5.7112	1.68
12	5.2545	1.54	54	5.7902	1.7	12	5.5270	1.62	54	5.4256	1.59
13	5.4579	1.60	55	5.6427	1.66	13	5.7970	1.70	55	5.5392	1.63
14	5.1646	1.52	56	5.8214	1.71	14	5.4253	1.59	56	5.6992	1.67
15	5.1090	1.50	57	5.5560	1.63	15	5.3248	1.56	57	5.5179	1.62
16	5.9207	1.73	58	5.5080	1.62	16	5.7506	1.69	58	5.8709	1.72
17	5.7042	1.68	59	5.7702	1.7	17	5.7235	1.68	59	5.4619	1.60
18	5.6544	1.66	60	5.7356	1.69	18	5.6989	1.67	60	5.2373	1.54
19	5.6327	1.65	61	5.6858	1.67	19	5.0724	1.49	61	5.4770	1.61
20	5.3973	1.59	62	5.9686	1.75	20	5.6890	1.67	62	5.3718	1.58
21	5.6976	1.67	63	5.3796	1.58	21	5.4439	1.60	63	5.6840	1.67
22	5.5067	1.62	64	5.6967	1.67	22	5.3330	1.57	64	5.8798	1.73
23	5.3110	1.56	65	5.4854	1.61	23	5.5055	1.62	65	5.7607	1.69
24	5.2868	1.55	66	5.4819	1.61	24	5.4780	1.61	66	5.8211	1.71
25	5.3565	1.57	67	5.5411	1.63	25	5.4692	1.61	67	5.5456	1.63
26	5.7860	1.7	68	5.5449	1.63	26	5.3900	1.58	68	5.4832	1.61
27	5.4476	1.6	69	5.7844	1.70	27	5.3195	1.56	69	5.5687	1.64
28	5.4200	1.59	70	5.3522	1.57	28	5.7995	1.70	70	5.5773	1.64
29	5.7243	1.68	71	5.5926	1.64	29	5.6795	1.67	71	5.4971	1.62
30	5.7336	1.68	72	5.5771	1.64	30	5.6989	1.67	72	5.5324	1.63
31	5.3353	1.57	73	5.6308	1.65	31	5.7363	1.69	73	5.5961	1.64
32	5.6735	1.67	74	5.3725	1.58	32	5.3658	1.58	74	5.6128	1.65
33	5.6182	1.65	75	5.6599	1.66	33	5.8227	1.71	75	5.6556	1.66
34	5.9083	1.74	76	5.3918	1.58	34	5.5620	1.63	76	5.5939	1.64
35	5.3836	1.58	77	5.7268	1.68	35	5.3155	1.56	77	5.6813	1.67
36	5.5427	1.63	78	5.4389	1.6	36	5.5297	1.62	78	5.4873	1.61
37	5.6693	1.67	79	5.7032	1.68	37	5.5917	1.64	79	5.4901	1.61
38	5.6100	1.65	80	5.7056	1.68	38	5.6569	1.66	80	5.5213	1.62
39	5.4830	1.61	81	5.3551	1.57	39	5.4227	1.59	81	5.4611	1.6
40	5.4354	1.60	82	5.6982	1.67	40	5.5251	1.62	82	5.6610	1.66
41	5.9145	1.74	83	5.2820	1.55	41	5.5989	1.64	83	5.3230	1.56
42	5.4404	1.60	84	5.4432	1.6	42	5.2519	1.54	84	5.4424	1.6

C  
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## HEAT OF COMBUSTION

## CSC COMPUTER SCIENCES CORPORATION

## INTEROFFICE CORRESPONDENCE

to B. Templeton, Manager  
Hazards Range*R. Ross*  
from R. Ross  
Chemist

date 06/26/79

subject Test Methods for Parr Bomb Calorimeter ~~and literature content~~

Ref: TWR # ARGGR1/2,75 Rocket Expulsion Charge

## I. Test Method Summary for Parr Bomb Calorimeter

## A) General Information:

Number of Tests - 3 on old, 3 on new - Total 6

## 1. Special Requirements:

Sample is analyzed in the bulk granular state.

## 2. Apparatus:

Parr Bomb Calorimeter

## B) Procedure

1. Standardization *PER ASTM D 240-64*  
*MIL-STD-268B, -method 302.1*  
Conducted per test method 117, 4.1<sup>1</sup>. Standard Benzoic Acid

2. Water Equivalent E = 2491.54

3. Heat of Combustion

Conducted per test method 117, 4.2<sup>1</sup>.

CHG #	EXP CHG WT (GRAMS)	+ Δ °C	Heat of Combustion(Calories/Gram)
20	1.0070	0.762	1885.4
23	1.0192	0.821	2007.02
99A	1.0115	0.828	2059.54
72	1.0163	1.202	2946.8
73	1.0153	1.198	2939.9
74	1.0121	1.193	2936.8

C  
O  
P  
Y

# AUTOIGNITION/DECOMPOSITION TEMPERATURES

Old sample No. 29			New sample No. 29		
Test No.	$\Delta T_A$	Autoignition	Test No.	$\Delta T_A$	Autoignition
	$^{\circ}\text{C}$			$^{\circ}\text{C}$	
1	156.2	138.7	1	150	135.5
2	186	180*	2	150	142.5
3	170.8	160	3	159	152
4	153.75	145	4	162.5	145.5
5	153.2	146	5	156.5	151
$\bar{X}$	164	147	$\bar{X}$	155.6	145
$\sigma$	12.7	7.8	$\sigma$	4.95	6

\* Bad test.

## Note:

1.  $\Delta T_A$  tests completed on the old material.
2. The decomposition temperature obtained on the old sample varied significantly.
3.  $\Delta T_A$  tests completed on the new material.
4. The decomposition temperature obtained on the new sample varied significantly.
5. There is a difference between the old and new material; the decomposition temperature for the new material is approximately  $7^{\circ}\text{C}$  lower.

## THERMAL STABILITY

### Summary of Thermal Stability Tests - 48 Hours at 75°C

A. Old -  $\sigma = 1.64$

Sample No.	Sample weight in	Sample weight out	Change in configuration	Change in weight	
	gm	gm		gm	%
10	5.5742	5.2115	None	0.3627	6.51

b. B. New -  $\sigma = 1.63$

Sample No.	Sample weight in	Sample weight out	Change in configuration	Change in weight	
	gm	gm		gm	%
6	5.5634	5.2661	None	0.2973	5.34

Note:

1. Samples were placed in oven at 0200 hours on 16 April 1979 at 75°C.
2. Samples were removed from oven at 0200 hours on 18 April 1979 and reweighed.

## WEIGHT LOSS

### Summary of Weight Loss - 4-Hour Test at 50°C, 18 Inches of Vacuum

#### A. Old material

Sample No.	Sample weight in	Sample weight after 4 hours	Weight loss	
	gm	gm	gm	%
5	5.2732	5.2319	0.0413	0.78
26	5.7860	5.7385	0.0475	0.82

#### B. New material

Sample No.	Sample weight in	Sample weight out	Weight loss	
	gm	gm	gm	%
2	5.2880	5.2537	0.0343	0.65
31	5.7363	5.7016	0.0347	0.60

### Summary of Weight Gain at Ambient Temperature and Humidity

#### A. Old material

Sample No.	Sample weight out oven	Sample weight after 66 hours	Weight gain	
	gm	gm	gm	%
5	5.2319	5.2575	0.0256	0.49
26	5.7385	5.7700	0.0315	0.55

#### B. New material

Sample No.	Sample weight out	Sample weight after 66 hours	Weight gain	
	gm	gm	gm	%
2	5.2537	5.2742	0.0205	0.39
31	5.7016	5.7204	0.0188	0.33

# WEIGHT LOSS (Contd)

## Summary of Weight Loss - 48-Hour Test at 50°C and 18 Inches of Vacuum

### A. Old material

Sample No.	Sample weight in	Sample weight out	Weight loss	
	gm	gm	gm	%
5	5.2575	5.1819	0.0756	1.43
26	5.7700	5.6773	0.0927	1.61

### B. New material

Sample No.	Sample weight in	Sample weight out	Weight loss	
	gm	gm	gm	%
2	5.2742	5.1817	0.0925	1.75
31	5.7204	5.6276	0.0928	1.62

## Summary of Total Weight Loss Combined - 4- and 48-Hour Test at 50°C and 18 Inches of Vacuum

### A. Old material

Sample No.	Sample weight in	Sample weight out	Weight loss	
	gm	gm	gm	%
5	5.2732	5.1819	0.0913	1.73
26	5.7860	5.6773	0.1087	1.33

### B. New material

Sample No.	Sample weight in	Sample weight out	Weight loss	
	gm	gm	gm	%
2	5.2880	5.1817	0.0858	1.62
31	5.7363	5.6276	0.0899	1.57

### Weight Loss

1. Samples were inserted into the oven at 1100 hours on 13 April 1979 at 50°C and 18 inches of vacuum.
2. Samples were removed at 1500 hours and reweighed - total time, 4 hours.
3. Samples were stored at ambient temperature and humidity for ~66 hours and then reweighed.
4. Samples were inserted into the oven at 0830 hours on 16 April 1979 at 50°C and 18 inches of vacuum.
5. Samples were removed at 0830 hours on 18 April 1979 and reweighed.
6. Results of the 4-hour test indicate that some moisture and/or volatiles were driven off.
7. The results of the 66-hour storage at ambient conditions indicate an affinity for moisture.
8. Results of the 48-hour test indicate that additional moisture and/or volatiles were driven off.
9. The accumulative total or effect of the old versus the new sample material indicates a difference in the amount of moisture and/or volatiles present between the old and new material.

## ELECTRICAL SPARK

### Summary of Electrical Spark Tests

#### A. Old sample No. 29

Test No.	Capacitance	Voltage level	Energy, $\frac{1}{2}CV^2$	Observations
	$\mu f$	v	joules	
1	1	1,000	0.5	No reaction
2	1	2,000	2	No reaction
3	1	3,000	4.5	No reaction
4	1	4,000	8	No reaction
5	1	5,000	12.5	No reaction
6	1	6,000	18	No reaction
7	1	7,000	24.5	No reaction
8	1	8,000	32	No reaction
9	1	9,000	40.5	No reaction
10	1	10,000	50	No reaction

#### B. New sample No. 29

Test No.	Capacitance	Voltage level	Energy, $\frac{1}{2}CV^2$	Observations
	$\mu f$	v	joules	
1	1	1,000	0.5	No reaction
2	1	2,000	2	No reaction
3	1	3,000	4.5	No reaction
4	1	4,000	8	No reaction
5	1	5,000	12.5	No reaction
6	1	6,000	18	No reaction
7	1	7,000	24.5	No reaction
8	1		32	No reaction
9	1		40.5	No reaction
10	1		50	No reaction

Note:

1. No reaction was noted at a 50-joule energy level for the old material.
2. No reaction was noted at a 50-joule energy level for the new material.
3. Ambient temperature was 75°F; relative humidity was 55%.



## FRICION PENDULUM

### Summary of Friction Sensitivity

Old material		New material	
Steel shoe	Fiber shoe*	Steel shoe	Fiber shoe
No reaction	NA	No reaction	NA
No reaction	NA	No reaction	NA
No reaction	NA	No reaction	NA
No reaction	NA	No reaction	NA
No reaction	NA	No reaction	NA
No reaction	NA	No reaction	NA
No reaction	NA	No reaction	NA
No reaction	NA	No reaction	NA
No reaction	NA	No reaction	NA
No reaction	NA	No reaction	NA

\* Fiber shoe tests are not conducted when there is no reaction from steel shoe results.

## IMPACT SENSITIVITY

### Summary of Impact Sensitivity Data

Old sample No. 22							New sample No. 3						
Test No.	3 3/4 Inches			10 Inches			Test No.	3 3/4 Inches			10 Inches		
	E	D	N	E	D	N		E	D	N	E	D	N
1			X	X			1			X	X		
2			X	X			2	X			X		
3	X			X			3			X	X		
4	X			X			4	X			X		
5	X			X			5	X			X		
6		X		X			6	X			X		
7		X		X			7	X			X		
8	X			X			8	X			X		
9			X	X			9	X			X		
10			X	X			10	X			X		

Old density =  $5.7217 - 0.2150 = 5.5067 / 3.40359 = 1.62 \text{ gm/cm}^3$  (polyethylene bag weight sub-  
 New density =  $5.7265 - 0.2783 = 5.4482 / 3.40359 = 1.60 \text{ gm/cm}^3$  (tracted from total weight)

1. A difference in impact sensitivity is detected – 40% explosion at 3 3/4 inch for the old material versus 80% at the same height for the new material.
2. An attempt is being made to establish the minimum height for a reaction on both samples.
3. The minimum drop height for the old material is 2 1/2 inches = 1.49 joules.
4. The minimum drop height for the new material is 2 1/4 inches = 1.12 joules.
5. No honest speculation as to the cause of the difference between the two samples as of yet. Will speculate something when chemical analysis and/or stability test results are in.
6. Reaction legend: E is explosion  
 D is detonation  
 N is no reaction

# SUMMARY OF OPEN-AIR BURN; PRECONDITIONED TO +145°F

Old material				New material			
Sample No.	Sample weight	Density	Burn time	Sample No.	Sample weight	Density	Burn time
	gm	gm/cm <sup>3</sup>	sec		gm	gm/cm <sup>3</sup>	sec
8	5.6338	1.66	0.8	9	5.5915	1.64	0.9
11	4.8269	1.42	1.1	10	5.3857	1.58	0.4
12	5.2545	1.54	0.9	11	5.4438	1.60	0.5
31	5.3353	1.57	1.1	12	5.5270	1.62	0.5
32	5.6735	1.67	1.1	13	5.7970	1.70	0.3
$\bar{X}$	5.3448	1.57	1.0	$\bar{X}$	5.5490	1.63	0.52
$\sigma$	0.3060	0.09	0.13	$\sigma$	0.1425	0.04	0.20

# SUMMARY OF OPEN-AIR BURN; NO PRIOR CONDITIONING (AMBIENT)

Old material				New material			
Sample No.	Sample weight	Density	Burn time	Sample No.	Sample weight	Density	Burn time
	gm	gm/cm <sup>3</sup>	sec		gm	gm/cm <sup>3</sup>	sec
34	5.9083	1.74	1.5	32	5.3658	1.58	1.2
35	5.3836	1.58	1.5	33	5.8227	1.71	0.9
36	5.5427	1.63	1.6	34	5.5620	1.63	0.8
42	5.4404	1.60	0.9	47	5.3997	1.59	0.8
43	5.2820	1.55	0.9	43	5.2494	1.54	1.0
44	5.5214	1.62	1.1	44	5.5341	1.63	0.6
45	5.7782	1.70	1.5	45	5.7935	1.70	0.5
46	5.3472	1.57	1.5	48	5.3555	1.57	0.4
$\bar{X}$	5.5255	1.62	1.3	$\bar{X}$	5.5103	1.62	0.8
$\sigma$	0.203	0.06	0.28	$\sigma$	0.20	0.06	0.25

# SUMMARY OF OPEN-AIR BURN; PRECONDITIONED TO -50°F

Old material				New material			
Sample No.	Sample weight	Density	Burn time	Sample No.	Sample weight	Density	Burn time
	gm	gm/cm <sup>3</sup>	sec		gm	gm/cm <sup>3</sup>	sec
50	5.3912	1.58	2.0	60	5.2373	1.54	0.5
51	5.3759	1.58	1.6	61	5.4770	1.61	0.4
52	5.2971	1.56	1.5	62	5.3718	1.58	0.3
53	5.8566	1.72	2.2	63	5.6840	1.67	0.8
54	5.7902	1.70	1.9	64	5.8798	1.73	2.0
$\bar{X}$	5.5422	1.70	1.8	$\bar{X}$	5.5299	1.63	0.8
$\sigma$	0.2328	0.068	0.3	$\sigma$	0.2279	0.07	0.6

# PRESSURE-TIME (-50°F CONDITIONED)

## Round-by-Round

### A. Old material

Sample No.	Sample weight	Density	Maximum pressure	Maximum rate rise	Maximum temperature	Gas volume	Burn time	Firing delay
	gm	gm/cm <sup>3</sup>	psi	psi/sec	°C	ℓ/gm	sec	ms
13	5.4579	1.60	47.87 46.15	66.80 67.54	373	2.82	0.88	82
14	5.1646	1.52	46.81 44.23	40.82 37.91	251	2.5	1.51	131
15	5.1090	1.50	47.87 46.15	89.76 89.32	427	3.27	0.62	172
28	5.4200	1.59	45.45 44.9	64.93 60	187	2.11	1.32	468
18	5.7042	1.66	45.74 45.19	68.61 66.13	229	2.17	0.78	236
$\bar{X}$	5.3711	1.57	46.04	65.18	293	2.57	1.02	218
$\sigma$	0.22	0.06	1.14	16.4	91.1	0.43	0.34	0.35

### B. New material

Sample No.	Sample weight	Density	Maximum pressure	Maximum rate rise	Maximum temperature	Gas volume	Burn time	Firing delay
	gm	gm/cm <sup>3</sup>	psi	psi/sec	°C	ℓ/gm	sec	ms
14	5.4253	1.59	56.82 55.10	252.53 236.14	290	2.08	0.52	68
15	5.3248	1.56	56.82 57.14	252.53 274.27	264	1.98	0.65	95
24	5.4780	1.61	59.09 59.18	253.24 257.30	316	2.04	0.53	89
17	5.7235	1.68	56.82 55.10	206.62 206.63	316	2.06	0.42	70
18	5.6989	1.67	56.82 55.10	166.30 194.47	316	2.07	0.83	108
$\bar{X}$	5.5301	1.62	56.80	230	300	2.05	0.59	86
$\sigma$	0.16	0.05	1.40	32.7	21	0.04	0.14	15

# PRESSURE-TIME (+145°F CONDITIONED)

## Round-by-Round

### A. Old material

Sample No.	Sample weight	Density	Maximum pressure	Maximum rate rise	Maximum temperature	Gas volume	Burn time	Firing delay
	gm	gm/cm <sup>3</sup>	psi	psi/sec	°C	ℓ/gm	sec	ms
55	5.6427	1.66	51.06 56.25	87.53 103.85	435	2.62	0.36	233
56	5.8214	1.71	55.32 63.54	115.65 131.46	316	1.91	0.63	137
57	5.5560	1.63	43.61 53.13	66.74 83.01	316	2.46	0.65	190
40	5.4354	1.60	53.19 64.58	103.62 122.23	285	1.95	0.75	133
41	5.9145	1.74	61.7 72.92	123.4 150.87	376	1.83	0.68	166
$\bar{X}$	5.6740	1.67	57.53	108.84	346	2.15	0.61	172
$\sigma$	0.17	0.05	7.9	23.79	53.6	0.32	0.13	37

### B. New material

Sample No.	Sample weight	Density	Maximum pressure	Maximum rate rise	Maximum temperature	Gas volume	Burn time	Firing delay
	gm	gm/cm <sup>3</sup>	psi	psi/sec	°C	ℓ/gm	sec	ms
35	5.3155	1.56	76.05 76.14	414.82 415.31	225	1.38	0.31	33
36	5.5297	1.62	80.28 77.27	437.89 441.54	225	1.28	0.47	58
37	5.5917	1.64	67.61 68.18	241.46 243.5	179	1.33	0.36	60
38	5.6569	1.66	76.06 75	325.97 310.34	255	1.38	0.31	50
39	5.4227	1.59	70.42 70.45	338.02 325.15	255	1.55	0.24	35
$\bar{X}$	5.5033	1.61	73.75	349.4	228	1.38	0.34	47
$\sigma$	0.12	0.04	4.05	71.14	27.8	0.1	0.08	11.3

# PRESSURE-TIME (AMBIENT CONDITIONED)

## Round-by-Round

### A. Old material

Sample No.	Sample weight	Density	Maximum pressure	Maximum rate rise	Maximum temperature	Gas volume	Burn time	Firing delay
	gm	gm/cm <sup>3</sup>	psi	psi/sec	°C	ℓ/gm	sec	ms
27	5.4476	1.60	50 48.07	102.04 99.46	315	2.47	0.77	144
2	5.5261	1.62	53.19 50	79.79 75	—	2.02*	0.54	79
3	5.6592	1.66	53.19 50.96	78.8 78.4	315	2.24	0.85	138
4	5.8154	1.71	51.06 48.08	65.18 67.94	162	1.69	1.18	99
6	5.7038	1.68	54.25 50.96	73.98 72.8	175	1.67	1.42	271
$\bar{X}$	5.6304	1.65	50.98	79.34	242	2.02	0.95	146
$\sigma$	0.130	0.04	1.98	11.6	85	0.35	0.31	67

### B. New material

Sample No.	Sample weight	Density	Maximum pressure	Maximum rate rise	Maximum temperature	Gas volume	Burn time	Firing delay
	gm	gm/cm <sup>3</sup>	psi	psi/sec	°C	ℓ/gm	sec	ms
1	5.3710	1.58	63.64 59.18	318.2 308.76	316	2	0.59	86
4	5.6362	1.66	61.36 59.18	266.78 273.14	363	2.1	0.43	99
5	5.7527	1.69	61.36 59.18	199.01 169.09	339	1.98	0.59	39
7	5.7645	1.69	55.68 54.08	133.63 135.2	316	2.09	0.59	94
8	5.6316	1.65	68.18 65.31	355.72 356.74	411	2.04	0.38	57
$\bar{X}$	5.6312	1.65	60.72	251.63	349	2.04	0.52	75
$\sigma$	0.142	0.04	4.03	82.06	35.5	0.05	0.10	23

# TIME FROM INITIATION TO PEAK PRESSURE

## Summary of Time to Peak Data from Pressure-Time Tests

Old pellets			New pellets		
Test No.	Sample conditioning	Time to peak*	Test No.	Sample conditioning	Time to peak
		ms			ms
A <sub>1</sub>	Ambient ~24°C	480 480	D <sub>1</sub>	Ambient ~24°C	200 190
A <sub>2</sub>	Ambient ~24°C	670 670	D <sub>2</sub>	Ambient ~24°C	220 230
A <sub>3</sub>	Ambient ~24°C	660 650	D <sub>3</sub>	Ambient ~24°C	310 350
A <sub>4</sub>	Ambient ~24°C	780 750	D <sub>4</sub>	Ambient ~24°C	420 400
A <sub>5</sub>	Ambient ~24°C	730 750	D <sub>5</sub>	Ambient ~24°C	190 180
B <sub>1</sub>	62°C	580 530	E <sub>1</sub>	62°C	180 180
B <sub>2</sub>	62°C	480 480	E <sub>2</sub>	62°C	180 180
B <sub>3</sub>	62°C	650 640	E <sub>3</sub>	62°C	280 280
B <sub>4</sub>	62°C	510 530	E <sub>4</sub>	62°C	230 240
B <sub>5</sub>	62°C	500 480	E <sub>5</sub>	62°C	210 220
C <sub>1</sub>	-45.6°C	720 680	F <sub>1</sub>	-45.6°C	230 230
C <sub>2</sub>	-45.6°C	1140 1160	F <sub>2</sub>	-45.6°C	210 230
C <sub>3</sub>	-45.6°C	520 500	F <sub>3</sub>	-45.6°C	230 230
C <sub>4</sub>	-45.6°C	700 710	F <sub>4</sub>	-45.6°C	280 270
C <sub>5</sub>	-45.6°C	670 680	F <sub>5</sub>	-45.6°C	340 280

\* Values are from two different strain-gage-type pressure transducers connected to the pressure vessel from a common "tee" at 90° angles.



C  
O  
P  
Y

## MOISTURE CONTENT

to B. Templeton, Manager  
Hazards Range

*R.P.*  
from R. Ross  
Chemist

date 06/20/70

subject Test Methods for Parr Bomb Calorimeter and Moisture Content page 2

### II. Test Method Summary for Moisture Content

- A) Number of Tests: 6
- B) Analysis Method: Karl Fischer Titration
- C) Sample is analyzed in the bulk granular state.
- D) Extraction solvent 70% Chloroform and 30% Methanol.
- E) Extraction solvent volume 125 ml
- F) Relative Humidity = 44%      Temperature 74°F
- F) Sample was soaked for 3 minutes prior to titration analytical note.

The water content of the extracting solvent was blanked out by Karl Fischer reagent. By subtracting out the water content of the solvent the water content of the sample is obtained by titrating the solvent + sample to an endpoint.

The moisture measurements are based on Karl Fischer Titration, in which the reagent reacts quantitatively with water in the sample to give a chemical change (titration endpoint) that is detected amperometrically.

H) Instrument: Beckman KF4B Aquameter

I) Standardization

Standardize with standard water - the water equivalence of STD H<sub>2</sub>O is in milligrams/milliliter (1 ml STD H<sub>2</sub>O = 1 mg H<sub>2</sub>O).

The water equivalence of Karl Fischer is determined as follows:

$F = A \times B / T$   
F = Water Equivalence of Karl Fischer reagent in mg/ml  
A = Milliliters of STD H<sub>2</sub>O used  
B = Water Equivalence of STD H<sub>2</sub>O  
T = Milliliters of Karl Fischer reagent required for titration

CSC

to B. Yempton, Manager  
Hazards Range

*R. Ross*  
from R. Ross  
Chemist

date 06/26/79

subject Test Methods for Parr Bomb Calorimeter and  
Moisture Content

page 3

Run #1 F = (1mgH<sub>2</sub>O/1 ml STD H<sub>2</sub>O) (10 ml STD H<sub>2</sub>O)/6.59ml KF

Run #1 F = 1.517

Run #2 F = (1mgH<sub>2</sub>O/1 ml STD H<sub>2</sub>O) (10 ml STD H<sub>2</sub>O)/6.51ml KF

Run #2 F = 1.536

Avg. Titer F = 1.53

#### J. Water Determinations

$$\frac{\text{H}_2\text{O ppm}}{\text{Molar}} = (\text{CF} \times 10^6)/W$$

C = Milliliters of Reagent (Karl Fischer) required to reach an endpoint

F = Water equivalence of Karl Fischer Reagent in mg/ml

10<sup>6</sup> = Factor for converting to parts per million

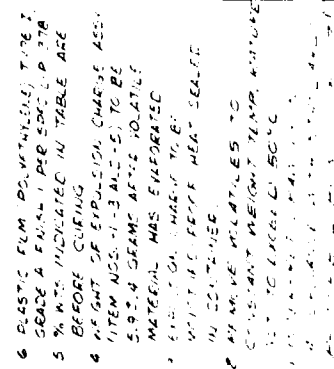
W = Weight of sample in milligrams

CHG #	Weight (mgs)	ml KF Reagent	H <sub>2</sub> O Content (Molar ppm)
33 old	1001.3	2.87	4.38 x 10 <sup>3</sup>
23 old	1019.2	3.62	5.43 x 10 <sup>3</sup>
99A old	1011.5	3.75	5.67 x 10 <sup>3</sup>
72 new	1016.3	1.53	2.30 x 10 <sup>3</sup>
73 new	1014.2	1.38	2.08 x 10 <sup>3</sup>
74 new	1012.1	1.02	1.54 x 10 <sup>3</sup>

CSC

COPY

**APPENDIX B**  
**DRAWING AND TABLES OF CHARACTERISTICS**



**UNITED STATES DISTRICT COURT  
SOUTHERN DISTRICT OF NEW YORK**

# TABLE OF CHARACTERISTICS

NOMENCLATURE M446 Expulsion Charge "OLD"

<b>COMPOSITION:</b>		<b>SENSITIVITY:</b>	
Ingredients:	Parts by wt.	Card Gap:	N/A
M9 Propellant	71.8	Detonation:	N/A
Black Powder FFF6(M12-P-223)	7.0	Electrical Spark:	> 50 Joules
Nitrocel Cement	14.2	Electrostatic:	
Potassium Chlorate/Baron (82.82/17.18)	7.0	Minimum Concentration	N/A oz/ft <sup>3</sup>
		Minimum Energy	Joules
<b>DRAWING NUMBER:</b>		Friction:	
65C11050		Steel Shoe	No reaction
<b>PARAMETRIC:</b>		Fiber Shoe	N/A
Auto Ignition Temperature	147 °C	Other	
Density		Ignition & Unconfined Burning	
Bulk Density	g/cm <sup>3</sup>	Single Cube	N/A Y N/A N N/A sec
Loading Density	1.62 g/cm <sup>3</sup>	Multiple Cube	Y N N sec
Decomposition Temp	°C	Impact Sensitivity	
Gas Volume		BoM	cm
Heat of Combustion	2.25 ± 0.44	PA	
Heat of Reaction	cal/g	BoE	2 1/2
Resistivity	ohm cm	Impingement	m/sec
Thermal Conductivity		<b>OUTPUT:</b>	
Watts/cm/degree		Burn Time	
Water Reactivity		Density 1.57	g/cm <sup>3</sup> 0.62 sec/cm
mi H <sub>2</sub> O Added: °C		Density 1.65	g/cm <sup>3</sup> 0.31 sec/cm
Fuel/Oxidizer Ratio		Density	g/cm <sup>3</sup> sec/cm
<b>STABILITY:</b>		Critical Diameter	
Hygroscopticity	95 %	Critical Height	
	90 %	Detonation Propagation	
	75 0.52 %	Wt	
	50 %	Density	g/cm <sup>3</sup>
Vacuum Stability		Velocity	m/sec
		Results	
		Pressure Time	
		psi/sec	84.5 ± 26
Thermal Stability		Time to Peak	msec 65 ± 17
	75°C 48 hrs	High Explosive Equivalency	
	Loss in wt 6.51%	P.A. Method	
Change in Configuration	None	Free Air Pipe Bomb	
Weight Loss	1.33	Closed Chamber	

### TABLE OF CHARACTERISTICS (Contd)

<b>APPLICATION &amp; ACCEPTANCE:</b>		<b>USE:</b> Expulsion charge for WOR-4A/A	
Luminous Output:	N/A	kilocandies	
Efficiency:	N/A	kilocandiesec/g	
Colorimetry:			
x coordinate			
y coordinate	N/A		
$\lambda$ (nm)			
Purity			
Burn Time:	0.86	0.34 sec	
<b>APPLICATION:</b>		<b>STORAGE:</b>	
Expulsion charge		Method:	Lry
		Hazard Class (O/D)	NATO 1.1      DoD 7
		Compatibility	G      A
<b>METHOD BLENDED:</b>		<b>METHOD LOADED</b>	
Wet blend		Pressed into 3.02 gm dia by 0.48 cm high pellets cured for 24 hours wt = 5.9 ±0.45 after cure, then sealed in moisture proof container.	
<b>REFERENCES/NOTES:</b>			

**TABLE OF CHARACTERISTICS (Contd)**  
**NOMENCLATURE**    **Expulsion Charge M446 "NEW"**

<b>COMPOSITION:</b> <div> <div>Ingredients:</div> <div>Parts by wt.</div> </div> M9 Propellant 71.8 Black Powder FFFG (MIL-P-223) 7.0 Nitrocel Cement 14.2 Potassium Chlorate/Boron (82.82/17.18) 7.0	<b>SENSITIVITY:</b>  Card Gap: N/A  Detonation: N/A  Electrical Spark: > 50 Joule  Electrostatic: <div> <div>Minimum Concentration</div> <div>N/A oz/ft<sup>3</sup></div> </div> <div> <div>Minimum Energy</div> <div>Joules</div> </div> Friction: <div> <div>Steel Shoe</div> <div>No reaction</div> </div> <div> <div>Fiber Shoe</div> <div>N/A</div> </div> <div> <div>Other</div> <div></div> </div> Ignition & Unconfined Burning:  <div> <div>Exploded</div> <div>Burn</div> </div> <div> <div>Single Cube</div> <div>Y</div> <div>N/A</div> <div>N</div> <div>N/A</div> <div>sec</div> </div> <div> <div>Multiple Cube</div> <div>Y</div> <div></div> <div>N</div> <div></div> <div>sec</div> </div> Impact Sensitivity:  <div> <div>BoM</div> <div>cm</div> </div> <div> <div>PA</div> <div>in</div> </div> <div> <div>BoE</div> <div>2 1/4</div> <div>in</div> </div> Impingement:  <div> <div>N/A</div> <div>m/sec</div> </div>
<b>DRAWING NUMBER:</b> 65C11050	
<b>PARAMETRIC:</b>  Auto Ignition Temperature: 145 °C  Density: <div> <div>Bulk Density</div> <div>g/cm<sup>3</sup></div> </div> <div> <div>Loading Density</div> <div>1.63 g/cm<sup>3</sup></div> </div> Decomposition Temp.: 156 °C  Gas Volume: 1.82 ± 0.33 ml/g  Heat of Combustion: cal/g  Heat of Reaction: cal/g  Resistivity: ohm-cm  Thermal Conductivity: Watts/cm/degree  Water Reactivity: ml H <sub>2</sub> O Added/ Δ°C  Fuel/Oxidizer Ratio:	<b>OUTPUT:</b>  Burn Time: <div> <div>Density</div> <div>1.55</div> <div>g/cm<sup>3</sup></div> <div>1.59</div> <div>sec./cm</div> </div> <div> <div>Density</div> <div>1.64</div> <div>g/cm<sup>3</sup></div> <div>0.5</div> <div>sec./cm</div> </div> <div> <div>Density</div> <div></div> <div>g/cm<sup>3</sup></div> <div></div> <div>sec./cm</div> </div> Critical Diameter:  Critical Height:  Detonation Propagation:  <div> <div>Wt</div> <div></div> </div> <div> <div>Density</div> <div></div> <div>g/cm<sup>3</sup></div> </div> <div> <div>Velocity</div> <div></div> <div>m/sec</div> </div> <div> <div>Results</div> <div></div> </div> Pressure Time  <div> <div>Time to Peak</div> <div>ps/sec 277 ± 85</div> <div>msec 25 ± 6</div> </div> High Explosive Equivalency P.A. Method Free Air Pipe Bomb Closed Chamber
<b>STABILITY:</b>  Hygroscopicity:  <div> <div>95</div> <div>%</div> </div> <div> <div>90</div> <div>%</div> </div> <div> <div>75</div> <div>0.36%</div> </div> <div> <div>50</div> <div>%</div> </div> Vacuum Stability:  <div> <div>ml/gas/40 hr</div> <div></div> </div> Thermal Stability:  <div> <div>75°C</div> <div>48 Hrs</div> </div> <div> <div>Loss in wt</div> <div>5.34%</div> </div> <div> <div>Change in Configuration</div> <div></div> </div> Weight Loss: 1.57 %	

**TABLE OF CHARACTERISTICS (Contd)**

<b>APPLICATION &amp; ACCEPTANCE:</b>		<b>USE:</b>	
Luminous Output:		Expulsion charge for WDU-4A/A	
Efficiency:	N/A kilocandles		
Colorimetry:	N/A kilocandlesec/g		
x coordinate			
y coordinate	N/A		
λ (nm)			
Purity			
Burn Time:	0.48± 0.16sec.		
<b>APPLICATION:</b>		<b>STORAGE:</b>	
Expulsion charge		Method: Dry	
		Hazard Class (O/D)	NATO DoD
			1.1 7
		Compatibility	G A
<b>METHOD BLENDED:</b>		<b>METHOD LOADED:</b>	
Wet: Blend		Pressed into 3.02 cm dia by 0.48 cm high pellets cured for 24 hrs. Wt=5.9 ±0.4 oz after cure, then sealed in moisture proof container.	
<b>REFERENCES/NOTES:</b>			



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